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SPECIFICATION FOR CYANURIC CHLORIDE

UDC 547·491·8'113·1 : 667·281/·285

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SPECIFICATION FOR CYANURIC CHLORIDE

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(*Continued on page* 2)

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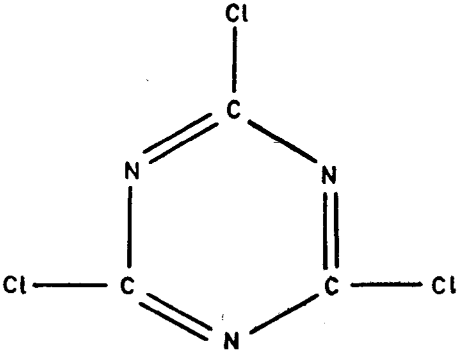
Indian Standard

SPECIFICATION FOR CYANURIC CHLORIDE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 27 July 1979, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Cyanuric chloride ($C_3N_3Cl_3$) is an important intermediate used in the manufacture of reactive dyes. It is also used in the manufacture of fluorescent brightening agents and herbicides. It is represented by the following structural formula:



CYANURIC CHLORIDE
(Molecular Mass 184·4)

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

---

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for cyanuric chloride.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of white or pale yellowish powder.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR CYANURIC CHLORIDE

Sl No.	Characteristic	Requirement	Method of Test, Ref to Appendix A
(1)	(2)	(3)	(4)
i)	Melting point, °C	145 to 147	A-1
ii)	Assay, percent by mass, *Min*	99·0	A-2
iii)	Free hydrochloric acid	To pass the test	A-3
iv)	Solubility in acetone	To pass the test	A-4

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in wide-mouthed epoxy-coated steel drums lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Batch number;

d) Gross, net and tare mass; and

e) The minimum cautionary notice worded as under:

> 'POISONOUS ! AVOID INHALATION AND CONTACT WITH SKIN AND EYES ' (vapour irritates the eyes and the mucous membrane).

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Test for assay shall be conducted on each of the individual samples.

4.2.2 Tests for the determination of remaining characteristics namely, melting point, free hydrochloric acid and solubility in acetone shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirement of assay if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (*see* **4.2.2**), the test results for each of the characteristics shall satisfy the relevant requirements given in Table 1.

5. TEST METHODS

5.1 Tests shall be carried out according to the methods prescribed in Appendix A. Reference to relevant clauses of Appendix A is given in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Methods of sampling and tests for dye intermediates.

†Specification for water for general laboratory use (*second revision*).

APPENDIX A

(*Table* 1 and *Clause* 5.1)

METHODS OF TEST FOR CYANURIC CHLORIDE

A-1. MELTING POINT

A-1.1 Determine the melting point of the material as prescribed in **8** of IS : 5299-1969*.

A-1.1.1 The melt should be clear and free from unmelted particles.

A-2. ASSAY

A-2.0 Outline of the Method — All the three hydrolysable chlorine atoms are reacted with a known excess of standard sodium hydroxide solution and the excess of sodium hydroxide is back-titrated.

A-2.1 Reagents

A-2.1.1 *Standard Sodium Hydroxide Solution* — 1 N.

A-2.1.2 *Standard Sulphuric Acid Solution* — 1 N (*see* IS : 266-1977†).

A-2.1.3 *Phenolphthalein Indicator Solution*

A-2.1.4 *Benzene*

A-2.1.5 *Sodium Hydroxide Solution* — 0·5 N.

A-2.1.6 *Sulphuric Acid* — 0·5 N.

A-2.2 Procedure

A-2.2.1 Weigh accurately about 1 g of the material into a stoppered conical flask. Add 40 ml of sodium hydroxide (1 N) solution and stopper the flask. Immerse the flask in a water bath maintained at about 50°C and shake it gently until all the crystals of cyanuric chloride are dissolved. Cool and titrate against sulphuric acid (1 N) using phenolphthalein as the indicator.

A-2.2.2 Weigh another 2 g of the same material into a separating funnel of 200 ml capacity. Add 30 ml of benzene and shake well to dissolve. Then add 100 ml of ice-cold water and shake well. Allow the layers to separate. Draw out the aqueous layer completely and filter it. Wash the filter with ice-cold water. Collect the filtrate and washings.

---

*Methods of sampling and tests for dye intermediates.

†Specification for sulphuric acid (*second revision*).

To this add 20 ml of sodium hydroxide solution (0·5 N) and shake well. Titrate the excess sodium hydroxide against sulphuric acid (0·5 N) using phenolphthalein as indicator.

A-2.3 Calculation

A-2.3.1 Total strength expressed as cyanuric chloride based on total acidity on hydrolysis $= \frac{(40 - A) \times 61 \cdot 5}{M \times 10}$

where

A = volume in ml of sulphuric acid (1 N) used in the titration, and

M = mass in g of the sample taken.

A-2.3.2 Free acidity (as HCl) $= \frac{(20 - B) \times 0 \cdot 5 \times 36 \cdot 5}{M_1 \times 10}$

where

B = volume in ml of sulphuric acid (0·5 N) used in the second titration, and

M_1 = mass in g of the sample taken.

A-2.3.3 Purity of cyanuric chloride $= X - (Y \times 1 \cdot 685)$

where

X = total strength expressed as cyanuric chloride based on total acidity on hydrolysis (*see* **A-2.3.1**), and

Y = free acidity (as HCl) (*see* **A-2.3.2**).

A-3. TEST FOR FREE HYDROCHLORIC ACID

A-3.1 Reagents

A-3.1.1 *Sodium Carbonate Solution* — 0·1 N.

A-3.1.2 *Methyl Orange Indicator Solution*

A-3.2 Procedure

A-3.2.1 Weigh accurately 5·0 g of the material and transfer to a 250-ml beaker containing ice-cold water and some ice. Stir at 0°C for about 20 minutes. Titrate with sodium carbonate solution using methyl orange indicator.

A-3.2.2 The material shall be taken to have passed if the volume of sodium carbonate solution required is not more than 2·0 ml.

A-4. TEST FOR SOLUBILITY IN ACETONE

A-4.1 Take about 2 g of the material in a test tube. Add 10 ml of acetone and shake well breaking the particles with glass rod.

A-4.1.1 The material shall be taken to have passed if the solution is clear and colourless.

AMENDMENT NO. 1 MAY 2002
TO
IS 9288 : 1979 SPECIFICATION FOR CYANURIC CHLORIDE

(*Page* 4, *clause* 2.2) — Insert the following new clause after **2.2:**

2.3 Particle Size — Not more than 2.0 percent by mass of the material shall be retained on 250 micron IS Sieve [IS 460 (Part 1) : 1985] and not more than 8.0 percent by mass of material shall be retained on 150 micron IS Sieve when tested in accordance with method given in **A-5.**

(*Page* 8, *clause* **A-4.1.1**) — Insert the following new clause after **A-4.1.1**:

A-5 PARTICLE SIZE

Transfer 25.0 g of the sample to the designated sieve and replace the lid. Start sieving gently by banging on a pad of latex foam or similar material. Brush the residue gently with a camel hair brush to ensure that any remaining soft aggregates are broken down, calculate and report the percentage retained on each sieve.

NOTE — Safety precautions as detailed for this test must be followed strictly. The test must be carried out in a fuming cupboard and rubber gloves, respirator and plastic apron must be worn. Base and sides of fuming cupboard must be lined with paper before commencing the test. After completion of test, transfer residue and the material passing through the sieves to a beaker containing about 1.5 litre of 10 percent sodium hydroxide solution. Mix well and allow to stand overnight in the fuming cupboard. Finally pour down the waste in sink followed by flow of cold water. Wipe the lining paper with a cloth soaked in 10 percent sodium hydroxide solution and screw up the paper in an outside dust bin.

A-5.1 Calculation

$$\% \text{ Retained on 250 micron sieve} = 4 \times X$$

where

X = g of sample retained on 250 micron sieve and

$$\% \text{ Retained on 150 micron sieve} = 4 \times (X + Y)$$

where

X = g of sample retained on 250 micron sieve, and
Y = g of sample retained on 150 micron sieve.

(PCD 11)

Reprography Unit, BIS, New Delhi, India